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Optimization of Palm Oil Boiler Ash Biomass Waste as a Source of Silica with Various Preparation Methods

Bunga Fisikanta Bukit[1,2], Erna Frida[3*], Syahrul Humaidi[3], Perdinan Sinuhaji[3], Nurdin Bukit[4]

[1] Doctoral Program Department of Physics, Universitas Sumatera Utara, 20155, Medan, Indonesia
[2] Universitas Quality Berastagi, Berastagi, Sumatera Utara 22152, Indonesia
[3] Department of Physics, Universitas Sumatera Utara, 20155, Medan, Indonesia
[4] Universitas Negeri Medan, Medan, Sumatera Utara 20221, Indonesia
* Corresponding author's e-mail: ernafridatarigan@usu.ac.id

ABSTRACT

Several studies have synthesized silica from waste. The silica synthesis method from agricultural waste aims to produce high purity silica with low contaminants at an affordable cost. This study synthesized silica from oil palm boiler ash (OPBA) by means of various methods, such as ball milling, coprecipitation, and modification with methyl trichlorosilane (MTCS). XRD characterization results showed that the OPBA synthesized with ballmill and coprecipitation method has the smallest particle size of 14.90 nm. Morphology showed the OPBA obtained by using the ballmill method, the OPBA synthesized with ballmill and coprecipitation method, as well as the OPBA synthesized with ballmill, coprecipitation, and modified with methyl trichlorosilane as spherical particles. At the same time, the FTIR results show an absorption peak which is a characteristic of silica confirmed by the XRF results, where silica content is dominant.

Keywords: silica, coprecipitation, ball milling, methyl trichlorosilane.

INTRODUCTION

The research on plantation and agricultural waste is currently being conducted. The use of high-value waste products can help control disposal and other related waste problems. One of the materials that can be produced from plantation waste is silica. Silica can be synthesized from plantation and agricultural wastes such as rice husk ash, corn husks, sugarcane bagasse, and palm oil boiler ash (OPBA) (Bukit et al., 2019; Gandhi et al., 2015; Roselló et al., 2015; Saharudin et al., 2018). Palm oil plantations and mills in Indonesia are overgrowing. Therefore, the waste generated is substantial. OPBA has not been widely applied, thus causing many environmental problems. OPBA contains SiO_2, which has the potential to be expanded as a geopolymer composite. The composition of SiO_2 compounds in OPBA is about 45.5–49.2% (Hamada et al., 2018). Silica is one of the most abundant compounds found in the world. Silica can be in gel, crystalline, or even amorphous form in nature, depending on how it is produced. (Todkar et al., 2016). Several previous studies synthesized silica from waste and used it as a coating, such as synthesis of silica from rice husk and modified with polydimethylsiloxane (PDMS), silica from rice husk ash modified with methyl trichlorosilane. In addition, silica nanoparticles from corn husk waste were synthesized through a leaching process with decomposition at high temperatures and by ball milling as well as modified with methyl trichlorosilane compounds to produce superhydrophobic solutions (Fahreza Harun, 2020; Martino, N, 2016; Shishodia et al., 2019).

Commercially high purity silica is quite expensive and unsuitable for most industrial

applications due to its high melting point (Anuar et al., 2020; Omar, Fen, & Matori, 2016; Omar, Fen, Matori, et al., 2016). Therefore, many methods have been explored to synthesize SiO_2 raw materials, as they are suitable for various applications and cost-effective simultaneously. The extraction concept and methodology of silica synthesis from agricultural waste and other derivatives mainly aims to produce high purity silica with low contaminants at affordable costs with higher prospects for large-scale production.

The environmental aspects of the synthesis process are also considered by improving conventional methods and replacing certain steps and chemicals with environmentally friendly ones (Osman & Sapawe, 2020). Silica synthesis from agricultural waste was carried out by means of several methods, such as sol-gel, combining acid leaching, chemical dissolution, as well as co-assembly with additional surfactants, calcination, mechanical milling, and coprecipitation (Bukit et al., 2018; Chun et al., 2020; Dang et al., 2018; Fisikanta et al., 2022; Ginting et al., 2020; Imoisili et al., 2020; Le et al., 2013). In this research, silica synthesis from oil palm boiler ash was carried out via varying methods such as ball mill, coprecipitation, and modification with methyl trichlorosilane.

EXPERIMENTAL DETAILS

Materials

OPBA from PT. DPI (Dhajaja Putra Indonesia) Asahan District North Sumatra Indonesia, 5M HCL, NH_4OH Merck Pro Analis, methyl trichlorosilane Sanghai Biotech, toluene.

Methods

Synthesis of SiO_2 nanoparticles from OPBA

The OPBA from palm oil mill waste was calcined at 500 °C for 5 hours, and then placed in a Planetary Ball Mill Retsch PM 200 for 10 hours with a speed rotation of 250 rpm. The milling process is dry milling with an effective sun wheel diameter of 157 mm. Materials of grinding tools are hardened steel, stainless steel, tungsten carbide, agate, sintered aluminum oxide, silicon nitride, zirconium oxide. Then, OPBA was mixed with 5M HCl in a ratio 1:4, stirred and heated at 70 °C for 4 hours using a magnetic stirrer with a speed rotation of 300 rpm and then filtered. Furthermore, OPBA was mixed with NH_4OH in a ratio of 1:4 for 4 hours at 70 °C with a speed rotation of 300 rpm (Bukit et al., 2019). The wet phase method, such as coprecipitation, is the best way to control particle size, shape and composition (Cargnello et al., 2014). Furthermore, OPBA was washed with distilled water to produce a neutral pH and dried at 70 °C for 4 hours. OPBA was modified with methyl trichlorosilane, using toluene as solvent. The variation of the ratio between OPBA and methyl trichlorosilane is 2:1. In addition, to make it easier to analyze the data, each sample is labeled, as shown in Table 1.

Characterization

X-ray diffraction analysis (XRD) was done using a Shimadzu 6000 goniometer. Characterization was carried out using Cu/Kα_1 radiation (λ = 1.54056 Å), Cu/Kα_2 radiation (λ = 1.54443 Å), Cu/Kβ radiation (λ = 1.39255 Å), an angle of 2θ from 7° to 70°, voltage was 40.0 kV, and current was 30.0 mA. The crystal size (D) of the material was determined using the equation:

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

Where: D is the crystal size (Å), λ is the wavelength of the X-ray used, κ is the Bragg angle, β is Scherrer's constant has a general value of 0.9 and is half the width of the Full Width at Half Peak Maximum (FWHM) in radians.

Morphology OPBA can be characterized by Scanning Electron Microscope (SEM). The SEM used is the SEM FEI Inspect S50 type. Before being characterized by SEM, the specimen was coated with metal. To determine the functional

Table 1. Sample label

No	Materials	Sample label
1	Raw OPBA	OPBA-R
2	OPBA ballmill	OPBA-B
3	OPBA ballmill and coprecipitation	OPBA-BC
4	OPBA ballmill, coprecipitation, and modified with methyl trichlorosilane	OPBA-BC-MTCS

group of OPBA, characterization was carried out using the FT-IR Shimadzu IR Prestige 21 made in Japan with Resolution = 4.0. Wavenumber range from 500 cm^{-1} to 4000 cm^{-1}. Furthermore, XRF characterization was carried out using XRF PANalytical Manipal 4. This tool can be used to test the elemental content of a material ranging from Sodium-Uranium.

RESULTS AND DISCUSSION

X-Ray diffraction (XRD) characterization

Crystal size was obtained using the analysis with the origin graph from equation one, as shown in Table 2. In addition, Table 3 shows the results of the XRD nanoparticle OPBA analysis. Boiler ash consists of quartz SiO_2 as the main crystalline phase at 2θ = 27°. The high intensity of the quartz phase is supported by the XRF analysis, where SiO_2 is the main content of OPBA.(Bukit et al., 2019; Zarina et al., 2013) The XRD pattern is shown in Figure1.

Scanning electron microscope (SEM) analysis

The morphology of OPBA in Fig. 2 is angular, irregular and crushed form with various

Table 2. Crystal size of sample

Sample code	Crystal size (nm)
OPBA-R	53
OPBA-B,	24.79
OPBA-BC	14.90
OPBA-BC-MTCS	30

Table 3. XRD analysis of OPBA-R, OPBA-B, OPBA-BC, OPBA-BC-MTCS

XRD data collection	OPBA-R	OPBA-B	OPBA-BC	OPBA-MTCS
Crystal system	Trigonal (hexagonal axes)	Trigonal (hexagonal axes)	Trigonal (hexagonal axes)	Triclinic (anorthic)
Space group	P 3121	P 312 1	P 32 2 1	P 1
Unit cell	a = 4.9019 Å c = 5.3988 Å	a = 4.9158 Å c = 5.4091 Å	a = 4.9230 Å c = 5.4090 Å	a = 4.9160 Å c = 5.4070Å, α=90°, ß=90°
Density	2.664g/cm³	2.664g/cm³	2.636 g/cm³	2.644 g/cm³

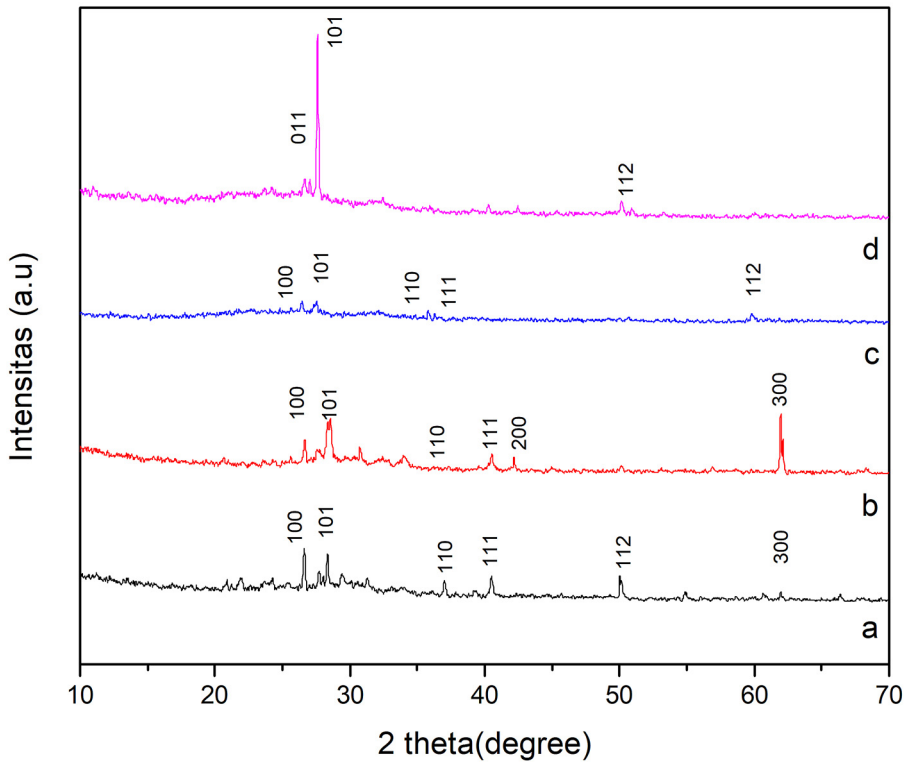

Fig. 1. XRD diffraction pattern of (a) OPBA-R (b) OPBA-B, (c) OPBA-BC, (d) OPBA-BC-MTCS

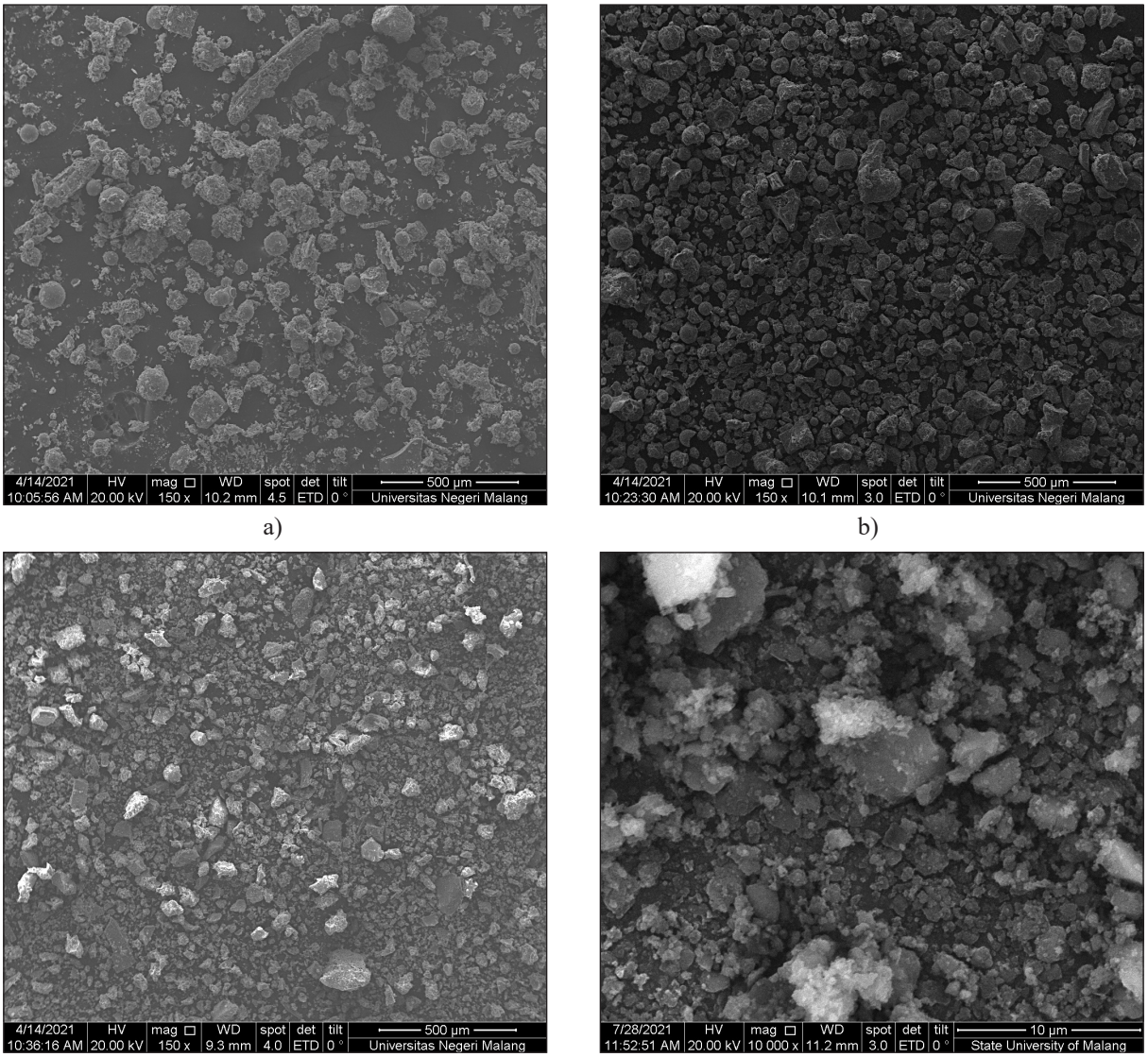

Fig. 2. The morphology of (a) OPBA-R (b) OPBA-B, (c) OPBA-BC, (d) OPBA-BC-MTCS

particle sizes. Heat treatment has changed the morphology of the boiler ash as observed under SEM. OPBA-R shows solid particles with non-uniform shapes, while heat treatment has changed the OPBA-B, OPBA-BC, and OPBA-BC-MTCS particles into spherical particles (Frida, Bukit, et al., 2022; Yahya et al., 2013).

Fourier transform infra-red (FTIR) analysis

FTIR spectrum of OPBA is shown in Fig. 3. In general, the absorption bands that appear do not have significant differences. The characteristic peaks around 993.0, 977.84, and 722.15 cm^{-1} are associated with asymmetric strain vibrations (Si-O-Si), in-plane strain vibration (Si-OH) and symmetrical strain vibration of (Si-O-Si), respectively (Rezaei et al., 2014). The signal at 1397.15-1668.18 cm^{-1} corresponds to the deformation of H_2O adsorbed between - silicate layers (Legarto et al., 2019). The absorption peak 3381.25-3590.90 cm^{-1} could be attributed to the hydroxyl vibration due to the hydroxyl ion (Melucci et al., 2019).

X-ray fluorescence (XRF) analysis

The chemical composition of OPBA after various treatments is shown in Table 4. The elements of OPBA change due to the different methods used. Dominant elements, such as SiO_2(Frida, Rahmat, et al., 2022), increased in OPBA-B and OPBA-BC. From the whole sample, it was found that OPBA-BC had the highest amount of SiO_2 and this is caused by the reaction of HCl with OPBA in the coprecipitation process

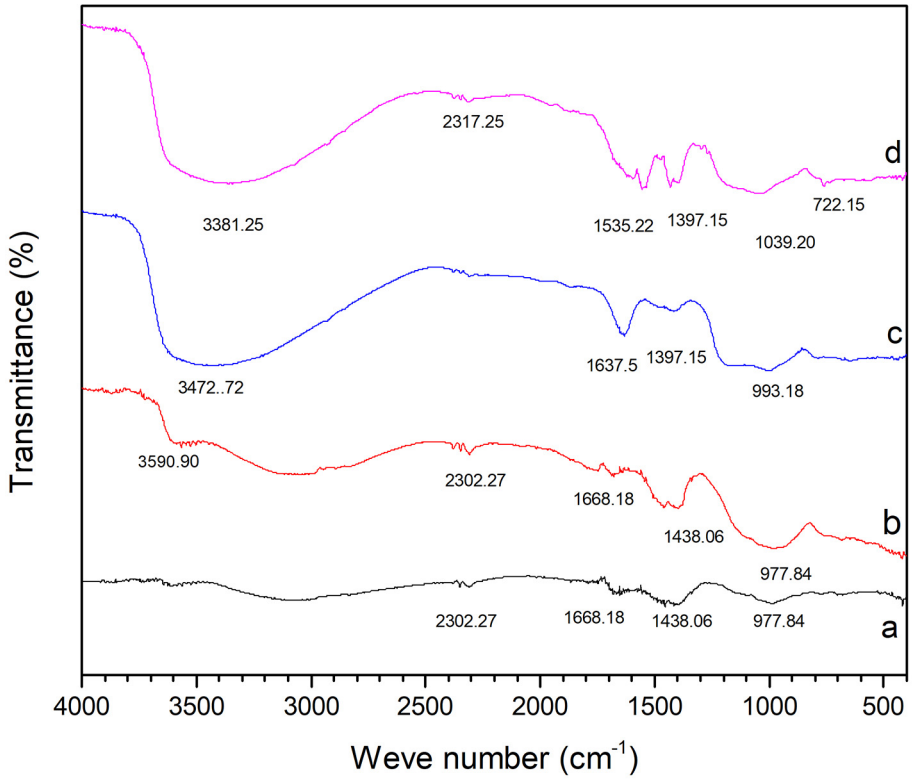

Fig. 3. FTIR spectrum of (a) OPBA-R (b) OPBA-B, (c) OPBA-BC, (d) OPBA-BC-MTCS

Table 4. Chemical composition of OPBA

Compound	OPBA-R	OPBA-B	OPBA-BC	OPBA-MTCS
SiO_2	27.3%	32.1%	48.5%	35.7%
Cao	16.1%	20.3%	23.6%	20.5%
K_2O	45.6%	34.6%	7.10%	7.82
Fe_2O_3	5.25%	5.97%	9.41%	7.60%
Cl	-	-	-	19.5%

(Iya et al., 2019). However, there was a decreas (Frida, Rahmat, et al., 2022)e in OPBA-MTCS. The presence of the Cl was due to the MTCS, which implied the existence of methyl groups (Gurav et al., 2015).

CONCLUSIONS

XRD characterization shows that OPBA-BC has the smallest crystal size of 14.90 nm. Morphology showed OPBA-B, OPBA-BC, and OPBA-BC-MTCS to be spherical particles. Meanwhile, the FTIR results indicate an absorption peak which is a characteristic of silica that also confirmed by the XRF results, where silica content is dominant.

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